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(54) Device and process for the direct introduction of samples into a mass spectrometer

(57) A device for the direct introduction of samples into a mass spectrometer comprises a direct introduction rod (1) incorporating sample holders (9) and adapted to introduce them sequentially into desorption zone (35) of an ionization chamber or ion source (not shown), a control mechanism (2) actuated by at least two stepping motors coupled to reduction gears for controlling the rod (1) and a sleeve (7) fixed in a dismantlable, tight manner to the end of the rod (1) and adapted to transfer sample holders (9) sequentially to the position for sample introduction into the ionization chamber or ion source. Sample holder introduction control is achieved by means of a programmable automaton or a microcomputer.

Introduction rod (1) may include supply rod (11) bearing on one end of the array of sample holders (9) in the rod, linearly reciprocable control rod (15) for forwarding a presented sample holder (9) to zone (35) and discharge rod (13) in sample holder discharge guide (12).

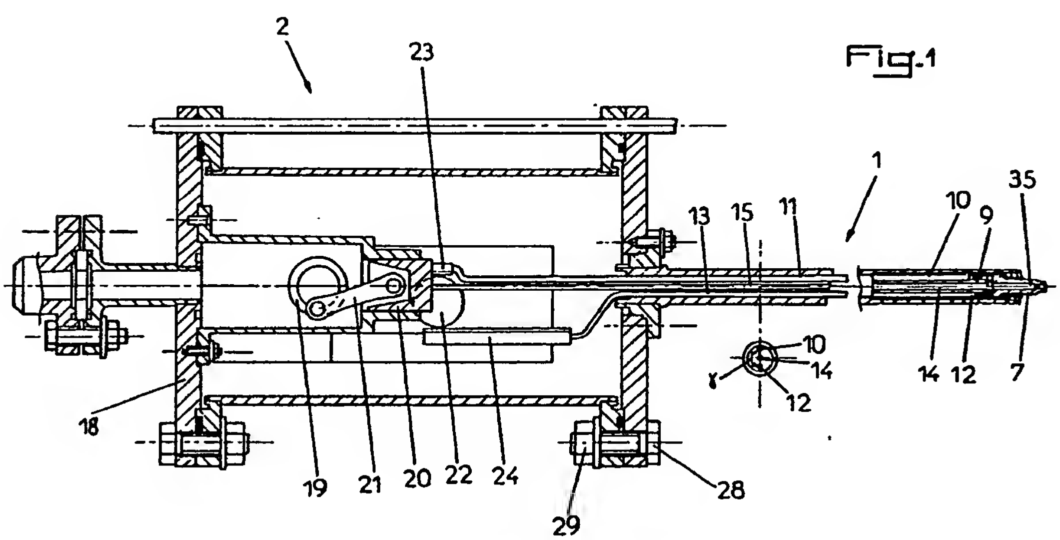

Fig.1

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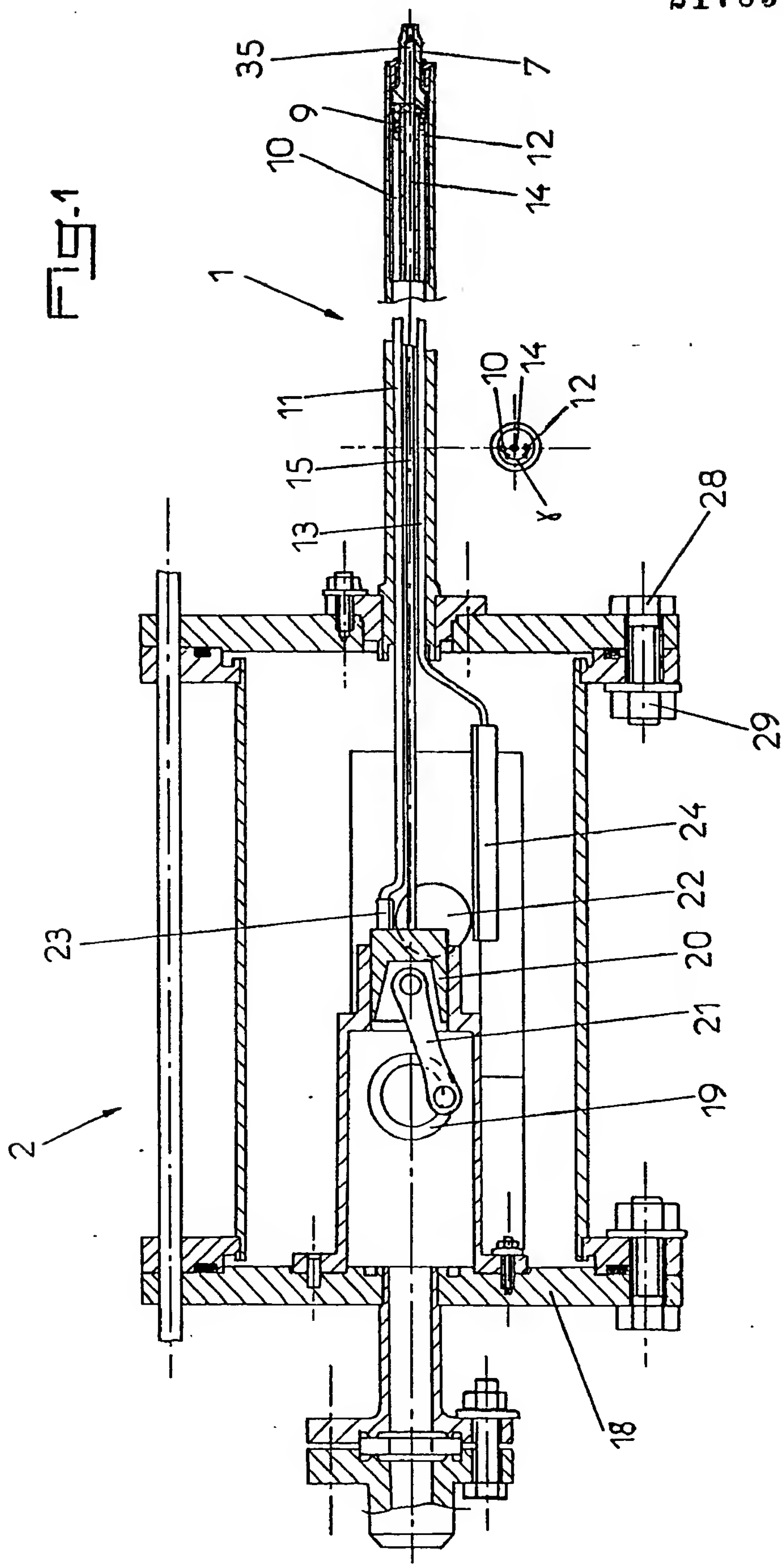
Fig.1



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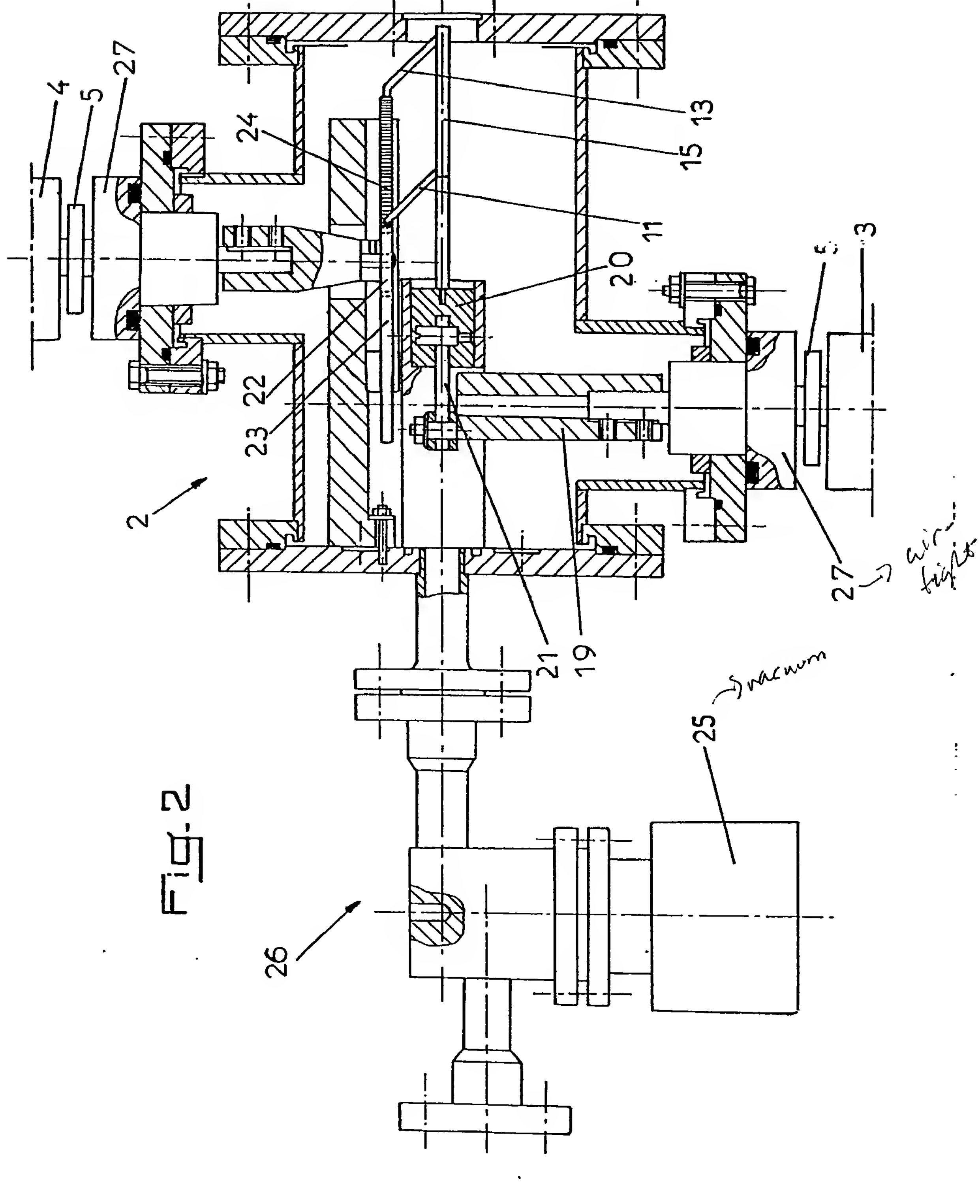

Fig.2

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Fig. 3

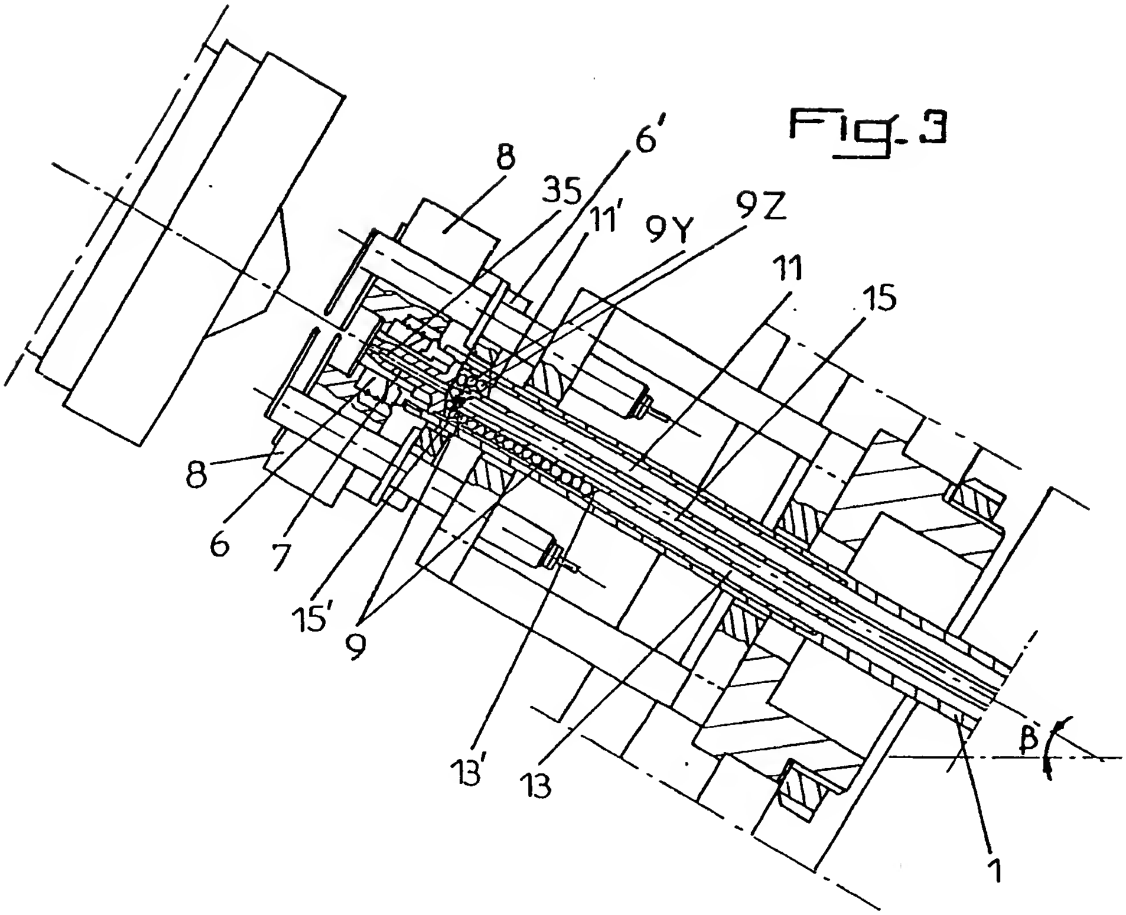


Fig. 6

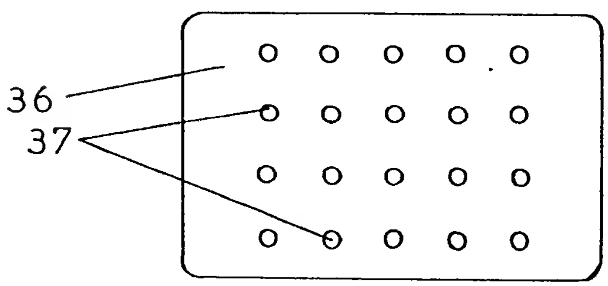


Fig-4

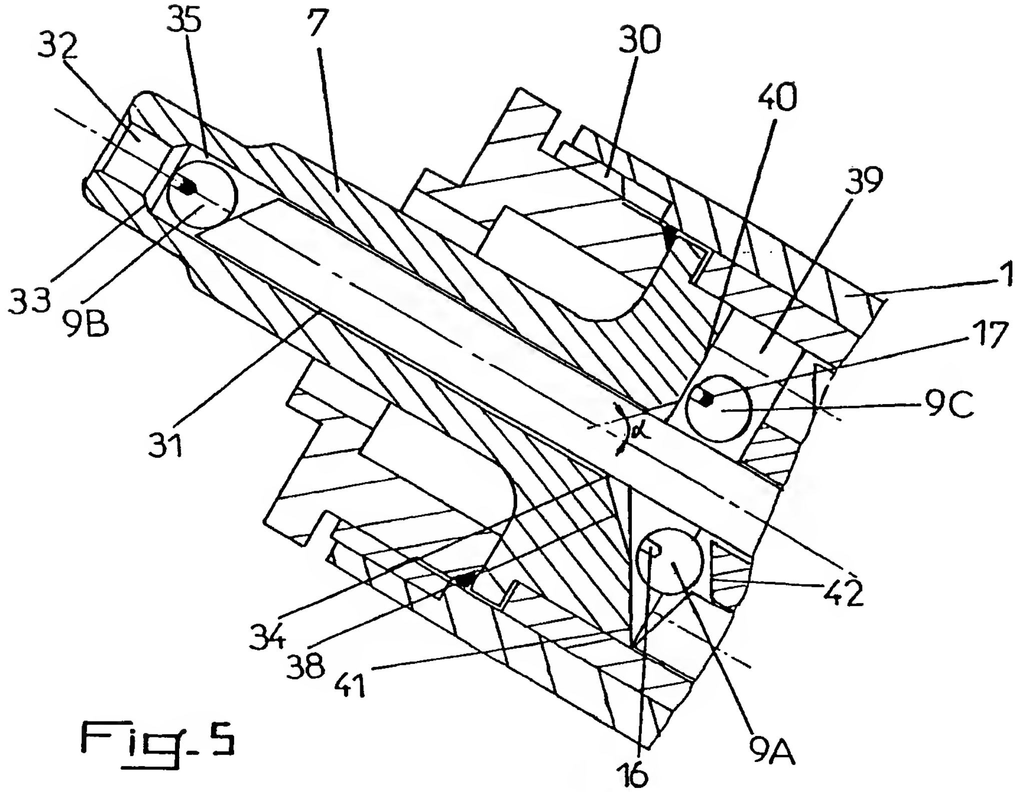
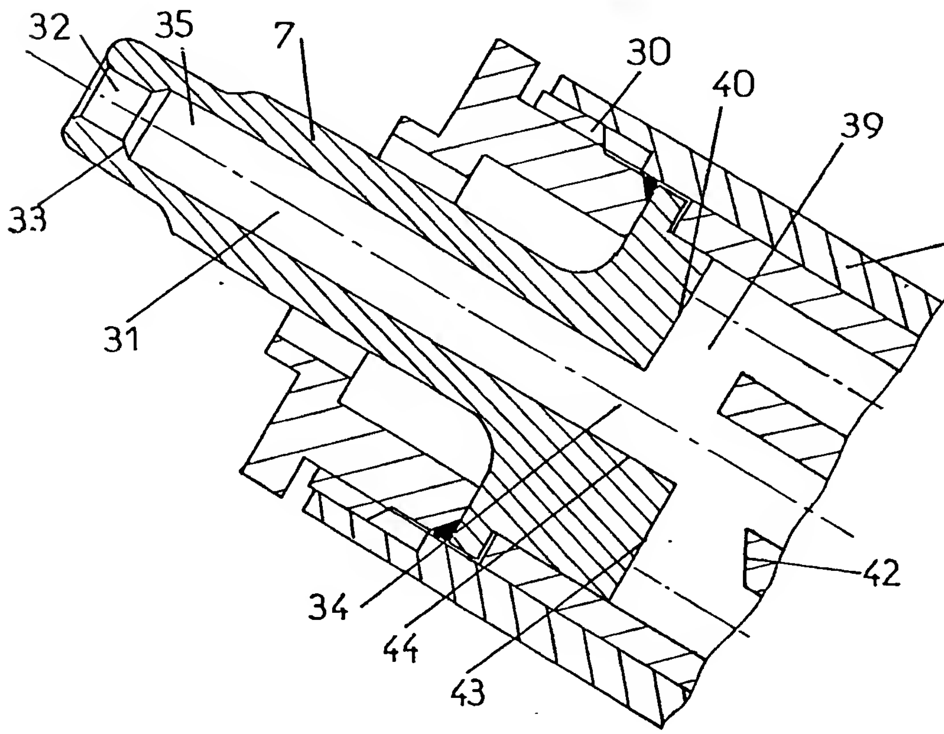

32
35
7
30
40
39
1
17
9C
33
9B
31
42
34
38
41
16
9A

Fig-5


32
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40
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31
42
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SPECIFICATION

Device for the direct introduction of samples into a mass spectrometer and process performed by this device

The present invention relates to the introduction of samples into a mass spectrometer and more specifically to a device for the direct introduction of samples Incorporating means for ensuring the successive continuous or intermittent introduction of several identical or different samples.

No existing device makes it possible to successively introduce in a continuous manner a numher of samples.

Sample introduction devices must make it possible to obtain a rapid scanning of the samples on the one hand for routine analyses, where in the case of slightly volatile, thermally unstable samples, e.g. peptides, desorption of the samples must take place in the ion source in the vicinity of the ionizing beam, and on the other hand for studies of the kinetics of ion molecule reactions, samples requiring total quantities exceeding those attainable by introduction in the conventional manner. The special feature of the introduction in the latter case is that the sample has to be introduced in a continuous manner for a relatively long period of time, so that the constant partial pressure of the sample in the source remains constant. The value of this partial pressure must be regulated by means of the total ion stream available at the analog output on control consoles of spectrometers.

However, the existing sample introduction devices, which are essentially placed in two classes, namely those of the carousel type and those of the rod type, suffer from the disadvantages referred to hereinafter.

In the case of an automatic supply by carousel, either outside or inside the source box, the disadvantages are due on the one hand to a problem of overall dimensions, because the space available in the source box is limited and on the other hand a considerable idle time between two samples makes it impossible to keep the partial pressure constant in the ion source. This second point is not vital for routine product analyses, but is incompatible with studies relating to the thermodynamics of ion—molecule reactions.

Thus, in the case of routine analyses, the absence of a solvent eliminates the peaks caused by the solvent product interaction and thus makes it possible to more easily define the molecular mass. However, it is obvious that th us of a solvent is incompatible with th determination of thermodynamic values and in this cas the ion—molecule system must be perfectly defined.

In the case of a supply by a rod, th advantage is directly linked with its shape, thus permitting an interchangeability of the rod as a function of the desired us , whilst readily adapting to different spectrometers.

However, the main difficulty is that the introduction of samples involves the presence of an operator, who has to introduce the rod carrying the sample up to the ion source. This introduction comprises cleaning the sample holder, putting the sample into place, opening the access valve, opening the access valve to the lock, introducing the rod Into the lock, closing the external lock valve, pumping into the lock, investigating the desired pressure in the lock, opening the ion source—lock valve, introducing the rod in such a way that the sample holder is in the ion source, heating the sample, taking the spectrum, removing the sample holder from the ion source, closing the lock access valve, cooling the rod and removing the rod from the lock.

These operations are required for the introduction of a single sample. Thus, e.g. if it is wished to introduce twenty samples, the aforementioned operating procedure has to be repeated twenty times, which involves a considerable time loss.

The present invention aims to obviate these disadvantages.

According to one aspect of the invention, there is provided a device for the direct introduction of samples into a mass spectrometer, in which sample holder introduction control is ensured by a sequential control by means of a programmable automaton or a microcomputer, wherein the device comprises a direct introduction rod incorporating a plurality of sample holders and adapted to introduce said sample holders sequentially into an ionization chamber or ion source, a control mechanism actuated by at least two stepping motors coupled to reduction gears for controlling the rod and a sleeve fixed in a dismantlable and tight manner to the end of the rod and adapted to transfer sample holders sequentially to a position in the rod for introduction into said ionization chamber or ion source.

Another aspect of the invention provides a process for the direct introduction of samples into a mass spectrometer, performed by the above-described device, in which the process comprises the steps of successively introducing in a continuous or intermittent manner for a time interval which is preset or settable as a function of the pressure measured in the ion source by a programmable automaton or a microcomputer, several identical or different samples into the vicinity of the ionizing beam of the ion source of the mass spectrometer, whilst optionally maintaining the partial pressure of the product constant in the ion sourc by succ ssive displacem nt of the sample holder with respect to th ion source and whilst optibnally varying the desorption time and desorption t mperatur b tween individual sampl s.

Th inv ntion will n w b further d scrib d,

by way of example, with reference to the drawings, in which:

Fig. 1 is a front sectional view of one embodiment of a direct introduction device according to the invention;

Fig. 2 is a part-sectional plan view of the control mechanism of the device shown in Fig. 1;

Fig. 3 is a section, to an enlarged scale, of the free end of the rod of the device which is to be introduced into the ionization chamber;

Figs. 4 and 5 are sections, to a still further enlarged scale, of variants of the transfer sleeve of the device according to the invention; and

Fig. 6 is a plan view of a sample filling support plate for use with the device according to the invention.

Referring to the drawings, the Introduction device according to the invention is mainly constituted by a device for the direct automatic introduction of samples into a mass spectrometer, wherein it is mainly constituted by a direct introduction rod 1 having all the sample holders 9 and these are introduced into an ionization chamber or ion source by a control mechanism 2 actuated by at least two stepping motors 3, 4 coupled to reduction gears 5 and by a transfer sleeve 7 for the sample holders 9, fixed in a dismantlable and tight manner to the end of the rod 1. These means are sequentially controlled, either by a programmable automaton or by a microcomputer, ensuring the direct introduction of the sample holders 9.

Thus, a single operation permits the successive introduction of several sample holders 9 with the aid of the rod 1 into the ionization chamber or ion source. In order to obtain the temperature necessary for the desorption of the product, the ionization chamber is provided with a heating device and a cooling device. The heating device is in the form of heating cartridges of a known type placed in a block 8 and supplied by a current between 1 and 2 Amperes. In the case of slightly volatile products, a complementary heating device in the form of a boron nitride heating collar 6 constituting an interface is placed within the ion source, so as to surround the sleeve 7 of the rod 1 introduced into the ion source and whilst being in contact therewith.

The cooling device is located outside the ion source and is in the form of a copper cooling collar 6′ forming an interface. The collar 6′ is cooled by a fluid, e.g. pressurized liquid nitrog n, which ciculat s in a channel placed in said collar.

Thus, a sample 17 is suddenly heated and desorption takes place v ry rapidly. Moreover, it is possibl to vary th temp ratur and desorption time b twe n individual sampl s 17.

As shown in Fig. 1, the direct introduction rod 1 contains sample hold rs 9 and has, on the on hand, at least one introduction guide 10 for the sample holders 9 in which slides a supply rod 11, whose free end 11′ has an inclined surface and, on the other hand, at least one discharge guide 12 for the sample holders 9 parallel to the introduction guide 10 and forming therewith an angle γ between 90° and 180°, in which slides a discharge rod 13, whose end 13′ also has an inclined surface. The two rods 11, 13 simultaneously perform a translation movement in opposite directions. Finally, there is a central guide 14 parallel to the introduction and discharge guides 10, 12 respectively, in which slides a central rod 15, whose free end 15′ also has an inclined surface having an alternating translation movement. The movement of the rods 11, 13 and 15 is produced by the control mechanism 2 connected in a fixed manner by bolts 28 and nuts 29 to the end of the rod 1 opposite to the ion source. Advantageously, all of the guides 10, 12 and 14 have an identical section slightly exceeding the diameter of the sample holder 9. The sample holders 9 are advantageously in the form of balls, each having at least one opening 16, which can contain a sample 17. The sample holders 9 are desirably made from stainless steel and advantageously have a diameter of approximately 2mm.

As shown in Figs. 1 and 2, the control mechanism 2 is constituted by a box 18 which contains, on the one hand, means making it possible to perform alternating translation movements of the central rod 15, said means being in the form of a crank 19 driven by the motor 3 and arranged to actuate a piston 20, to which is rigidly fixed the central rod 15, in an alternating translation movement, by means of a link 21, and, on the other hand, means permitting the performance of simultaneous translation movements in opposite directions of the supply rod 11 and discharge rod 13. These latter means comprise a gear wheel 22 driven by the motor and arranged to drive two diametrically opposite racks 23, 24. The control mechanism 2 is connected to a vacuum pump 25 via an isolating valve 26, the sealing of the transmission of the rotary movement of the two stepping motors 3, 4 located outside the mechanism 2 to the gear wheel 22 and crank 19 being ensured by two air-tight rotary passages 27.

Thus, the control mechanism 2 ensures the displacement of the supply and discharge rods 11, 13 respectively, as well as the central rod 15. The displacement of the supply and discharge r ds takes place by means of the gear whe l 22, which drives the racks 23, 24 connected to the rods 11 and 13, the rack 23 being located abov th gear wh el 22 and th rack 24 below the gear wh el 22, th rotation of the latter, controlled by th motor 4, driving each of the racks 23, 24 in an opposite direction and, therefore, the supply and discharg rods 11, 13 in a simultaneous

translation movement, but in opp site directions. The central rod 15 is displaced by means of the crank 19 driven by the motor 3, the rotary movement of the crank 19 being converted into a translation movement by the link 21 and piston 20, the central rod 15 being welded to the piston 20.

As shown in Fig. 4, the cylindrical transfer sleeve 7 is fixed in a dismantlable and tight manner to the end of the rod 1 remote from the control mechanism 2 by means of a threaded bush 30. The sleeve 7 is provided with a concentric cylindrical channel 31 having a diameter slightly exceeding the diameter of a sample holder 9 in the form of a ball, so as to permit the central rod 15 to introduce and maintain in place the sample holder 9, whose sample is to be desorped in said concentric cylindrical channel 31. The latter is provided at its free end 32 with a slightly smaller section than that of a sample holder 9, so as to form a stop 33 for said sample holder in the desorption zone 35 within the ionization chamber or ion source, whilst at its opposite end 34 it has a widened frustoconical section 38, the angle α of the frustum being between 30° and 60°, said widened section 38 being connected to a transfer slot 39 extending in front of the parallel guides 10, 12 and 14, being open from the side of the introduction guide 10 and having a rectilinear side face 40 facing introduction guide 10 and an inclined side face 41 facing the discharge guide 12. At its outlet, the latter is also provided with an inclined side face 42 parallel to the inclined side face 41, the spacing between these two faces 41, 42 being equal to the section of the guides 10, 12 and 14.

As a result of the specific geometry of the transfer sleeve 7, it is possible to successively introduce the sample holders 9 into the ionization chamber solely by the translation of the different rods 11, 13 and 15 within the rod 1.

According to another feature of the invention, the introduction device is inclined with respect to the horizontal plane and forms with the latter an angle β of at least 10°, the control mechanism 2 being in the lower position with respect to the end of the rod 1 remote from said control mechanism 2, and the introduction guide 10 is located in the upper part of the rod 1 above the central guide 14, which is itself above the discharge guide 12.

This inclination of the rod 1 with respect to the horizontal plane will obviously be a function of the position and inclination of the ionization chamber in the mass spectrometer.

In order that the first sample holder 9B in th form of a ball to be introduced into th concentric channel 31 of th transfer sl v 7 is position d exactly facing the central rod 15 and does not drop by gravity into the discharg guid 12, it is n c ssary to introduc beforehand an empty sample holder 9A into the discharg guide 12 against the discharg rod 13, so as to maintain the first sample holder 9B in place. Thus, the sample holder 9A, which will not be introduced into the desorption zone 35, will be in contact with the discharge rod 13 and with the first sample holder 9B to be introduced, which is itself in contact with the preceding sample holder 9A and the following sample holder 9C and so on up to sample holder 9Z, which will be in contact with the preceding sample holder 9Y and supply rod 11.

It is possible to obviate the prior introduction of an empty sample holder 9A into discharge guide 12, the sample holder 9B then being maintained in place in front of the concentric channel 31 by the positioning of central rod 15 in such a way that part of the end 15' of the central rod 15 is located in the transverse groove 39.

According to a variant of the invention not shown in the drawings, the transfer sleeve 7 is provided on its rectilinear side face 40 with retractable stop in the form of a push-button or a deformable metal blade. This variant is particularly suitable in the case where each of the rods 11, 13, 15 is independently controlled by a stepping motor, or in the case of a rod 1 having several introduction guides, each of the supply rods, together with the central rod and discharge rod being independently controllable by a stepping motor.

According to another variant of the invention shown in Fig. 5, the transfer sleeve 7 is provided with a concentric cylindrical channel 31 of a diameter slightly exceeding that of a sample holder 9 in the form of a ball, so as to permit the central rod 15 to introduce and maintain in place the sample holder 9, whose sample is intended for desorption in said concentric cylindrical channel 31. The latter is provided at its free end 32 with a section slightly smaller than that of a sample holder 9, so as to form a stop 33 for the sample holder 9 in the desorption zone 35 located within the ionization chamber or ion source, whilst at its other end 34 it has a transverse groove 39 extending in front of the introduction guide 10 and central guide 14, being open from the side of the introduction guide 10 and having an end 44 forming a stop in the extension of the concentric channel 31, as well as a first rectilinear side face 43 facing the introduction guide 10 and a second rectilinear side face 43 facing the discharge guide 12 and displaced with respect to the first side face 40. At its outlet, the discharge guide 12 is provided with an inclined side face 42, the displacement between the two side faces 42, 43, as well as the width of the transvers groov 39 being at least qual to the section of the guides 10, 12 and 14.

Thus, the sampl holder 9B is maintained in place pr cisely facing the conc ntric channel 31, whilst resting against th nd 44 of th groove 39 locat d in the ext nsion of th

concentric channel 31 and thus forming a stop.

The invention also relates to a process for the direct introduction of samples performed by the introduction device shown in Fig. 1. This process consists of bringing about the successive introduction, in a continuous or intermittent manner for a time interval which is pre-set or pre-settable as a function of the pressure measured in the ion source by a programmable automaton or a microcomputer, of several identical or different samples 17 in the vicinity of the ionizing beam of the ion source of the mass spectrometer, whilst optionally maintaining the partial pressure of the product constant in the ion source by successive displacement of the sample holder 9 with respect to said ion source and whilst optionally varying the desorption time and desorption temperature between the individual samples 17.

The following operations take place under the control of the programmable automaton or microcomputer:

manual deposition of a solid sample or a solution 17, which is thermally stable or unstable, in the opening 16 provided in each sample holder 9;

manual elimination of the solvent in the case of a sample in solution;

manual introduction of the sample holder 9 into the introduction guide 10 of rod 1 with the aid of a carrying gripper, the opening 16 of each sample holder 9 being oriented towards the transfer sleeve 7;

manual introduction of the free end of the rod 1 into the ionization chamber through a lock;

heating the desorption zone 35 within the ionization chamber 6;

checking the temperature;

heating or cooling the desorption zone 35 until the desired temperature t_1 is obtained;

putting the first sample holder 9B in place in the desorption zone 35;

desorption in the vicinity of the ionizing beam of the ion source of the mass spectrometer for a time interval dt;

optionally, checking the pressure in the ion source, the sample holder 9 remaining in the desorption zone 35 until the pressure tends to decrease;

removal of the first sample holder 9B;

checking the temperature;

heating or cooling the desorption zone 35 until either temperature t_1 or temperature t_2 is obtained, temperature t_2 being higher or lower than t_1;

placing th s cond sample holder 9C in the desorption zone 35;

continuing these operations until the final sampl holder 9Z is plac d in th desorption zone 35;

d sorption of the final sample holder 9Z in the vicinity of the ionizing beam of the ion source of the mass spectromet r for a time interval dt; and

removing the final sample holder 9Z.

The desorption zone 35 is heated or cooled with the aid of the heating collar 6 and cooling collar 6' described hereinbefore. Thus, prior to the introduction of each sample holder 9 into desorption zone 35, it is possible to vary the desorption temperature and also the desorption time. The maximum temperature to be reached in the desorption zone 35 is approximately 900°C. The time interval during which each sample holder 9 remains in the desorption zone 35 is a function of the nature of the samples 17. It varies from a few seconds to several hours.

For kinetic studies of ion—molecule reactions, a sample 17 has to be introduced into the desorption zone 35 for a relatively long time interval, so as to obtain a constant partial pressure of the product in the ion source. The value of this partial pressure must be regulated by means of the total ion stream available at the analog output on the control consoles of the spectrometer. This pressure is max approximately 1 Torr.

In order to deposit a solid or liquid sample 17 in an opening 16 of a sample holder 9, the following stages are manually performed:

placing sample holders 9 in the form of balls on a metal support plate 36 having semicylindrical recesses 37 (see Fig. 6), each of which is able to receive a ball-type sample holder 9 with the aid of a carrying gripper, the opening 16 of each sample holder 9 being oriented upwards; and

deposition of a liquid or solid sample 17 in the opening 16 of each sample holder 9 with the possibility of depositing different samples 17 in individual sample holders 9.

In order to eliminate the solvent in the case of samples in solution which are thermally stable, the following stages are manually performed.

introduction of the support plate 36 into a vacuum enclosure connection to a primary pump on a heating plate located in said vacuum enclosure until the solvent is eliminated;

heating the support plate 36; and

possible reduction of the pressure in the vacuum enclosure containing the sample holders 9 and the heating plate, in order to accelerate the elimination of the solvent.

To eliminate the solvent in the case of samples in solution of a thermal unstable nature, the following stages are manually performed:

introduction of the support plate 36 into a vacuum enclosure connected to a primary pump; and

reduction of the pressur in th vacuum nclosure containing the sample holders until the solv nt is eliminat d.

To simultaneously perform th putting into place of a sample holder 9C and the discharge of a pr ceding sampl holder 9B with r spect

to the desorption zone 36 and in th case where an empty sample holder 9A has been previously introduced into the discharge guide 12, the following stages are performed under the control of the programmable automaton or the microcomputer:

starting up the motor 3 when the desorption of the sample in sample holder 9B has taken place, motor 3 controlling the central rod 15 which drops, sample holder 9B thus being again transferred into the transverse groove 39 against sample holder 9A;

stopping motor 3;

starting up motor 4 controlling the translation of supply rod 11, which moves sample holders 9 and in particular sample holder 9C, the latter also moving sample holder 9B and takes up position in front of the inclined end 15' of central rod 15, due to the simultaneous translation of discharge rod 13, which enables the sample holder 9B to enter discharge guide 12;

stopping motor 4;

starting up motor 3 controlling the central rod 15, which introduces sample holder 9C into the channel 31 of transverse sleeve 7 until it strikes against stop 33; and

stopping motor 3.

In order to simultaneously perform the putting into place of a sample holder 9C and the discharge of a preceding sample holder 9B with respect to the desorption zone 35, in the case where no empty sample holder 9A has previously been introduced into the discharge guide 12 and where the transfer sleeve 7 has an inclined side face 41, the following stages are performed under the control of the programmable automaton or microcomputer:

starting up motor 3 when the desorption of sample 17 in sample holder 9B has taken place, the motor 3 controlling the central rod 15 which drops, the sample holder 9B thus dropping by gravity into the discharge guide 12 against the end 13' of the discharge rod 13;

stopping motor 3 when part of the end 15' of the central rod 15 is in the transverse groove 39;

starting up motor 4 controlling on the one hand the translation of the supply rod 11, which moves the sample holders 9 and in particular sample holder 9C, which drops by gravity into the transverse groove 39 against the end 15' of the central rod 15 and on the other hand the translation of the discharge rod 13, so as to create a space in the discharge guide 12 making it possible to receive the sample hold r 9C after desorption;

stopping motor 4;

starting up motor 3 controlling th central rod 15, which introduces th sampl holder 9C into the channel 31 of th transf r sl eve 7 up to contact with th stop 33; and

stopping motor 3.

In rd r to simultaneously carry out the putting into plac of a sample holder 9C and the discharge of the preceding sample holder 9B with respect to the desorption zone 35, in the case where no empty sample holder 9A has previously been introduced into the discharge guide 12 and in the case where the transfer sleeve 7 has a rectilinear side face 43 and an end forming a stop 44, the following stages are performed under the control of the programmable automaton or the microcomputer;

starting up motor 3 when the desorption of a sample 17 in the sample holder 9B has taken place, the motor 3 controlling the central rod 15 which drops, so that the sample holder 9B drops by gravity into the discharge guide 12 against the end 13' of the discharge rod 13 and then rises again until aligned with the rectilinear side face 43;

stopping motor 3;

starting up motor 4 controlling on the one hand the translation of the supply rod 11, which moves the sample holders 9 and in particular the sample holder 9C, which drops by gravity into the transverse groove 39 against the end 44 of said groove 39 and on the other hand the translation of the discharge rod 13, so as to produce a space in the discharge guide 12 making it possible to receive the sample holder 9C after desorption.

stopping motor 4;

starting up motor 3 controlling the central rod 13, which introduces the sample holder 9C into the channel 31 of the transfer sleeve 7 until it strikes the stop 33; and

stopping motor 3.

Finally, the programmable automaton or microcomputer also controls the display of measured digital values, printing by printer and alarm and safety devices.

The invention is not limited to the embodiments described and shown in the drawings but modifications may be made, particularly from the standpoint of the construction of the various parts, or by the substitution of technical equivalents, without passing beyond the protective scope of the invention.

CLAIMS

1. A device for the direct introduction of samples into a mass spectrometer, in which sample holder introduction control is ensured by a sequential control by means of a programmable automaton or a microcomputer, wherein the device comprises a direct introduction rod incorporating a plurality of sample holders and adapted to introduce said sample holders sequentially into an ionization chamber or ion source, a control mechanism actuated by at least two st pping motors coupled to reduction gears for controlling th rod and a sleeve fixed in a dismantlabl and tight mann r to th end of th rod and adapted to transfer sample holders sequ ntially to a position in th rod for introduction into said ionization chamb r or ion source.

2. An introduction device according to claim 1, wherein the direct introduction rod includes at least one introduction guide for the sample holders in which a supply rod is slidably mounted, the free end of said supply rod having an inclined surface; at least one discharge guide for the sample holders which extends substantially parallel to the introduction guide and forms therewith an angle between 90 and 180°, a discharge rod being slidably mounted in the discharge guide and having an end which also has an inclined surface, the supply rod and discharge rod simultaneously performing a translation movement in opposite directions; and a central guide extending substantially parallel to the introduction and discharge guides in which a central rod is slidably mounted according to an alternating translation movement, the central rod having a free end which also has an inclined surface; movement of said rods being produced by the control mechanism which is connected in a fixed manner to the end of the direct introduction rod remote from the ionization chamber or ion source and the guides each having a cross-section slightly exceeding the diameter of a sample holder.

3. An introduction device according to claim 2, wherein the sample holders are in the form of balls, each having at least one opening for containing a sample.

4. An introduction device according to claim 3, wherein the sample holders are made from stainless steel and have a diameter of approximately 2mm.

5. An introduction device according to any one of claims 2 to 4, wherein the control mechanism comprises a box which contains means enabling the central rod to perform alternate translation movements, said means taking the form of a crank driven by a first stepping motor located outside the box and actuating a piston which is connected to the central rod by means of a link, and means permitting the performance of simultaneous translation movements in opposite directions of the supply rod and discharge rod, said means taking the form of a gear wheel driven by a second stepping motor which is also located outside the box, the gear wheel being arranged to drive two diametrically opposite racks.

6. An introduction device according to claim 5, wherein the control mechanism is connected to a vacuum pump via an isolating valve, the sealing of the transmission of the rotary movement of the two stepping motors locat d outside the box to th crank and gear wheel being ensured by two tight rotary passages.

7. An introduction device according to any one of claims 2 to 6, wherein the transfer sleeve is provid d with a concentric cylindrical channel having a diameter slightly xce ding th diam t r of a sample holder, so as to enable the central rod to Introduce and maintain in place the sample holder, whose sample is intended for desorption in the concentric cylindrical channel, the latter having at its free end a cross-section slightly smaller than that of the sample holder, so as to form a stop for the sample holder in a desorption zone located within the ionization chamber or ion zource, and, at its other end, a widened cross-section in the form of a frustum of a cone, the angle of the cone frustum being between 30 and 60°, said widened cross-section being connected to a transverse groove extending in front of the Introduction, discharge and central guides, being open on the side of the introduction guide and having a rectilinear side face facing the introduction guide and an inclined side face facing the discharge guide, the latter also being provided at its outlet with an inclined side face parallel to the above-mentioned inclined side face and the space between the said inclined side faces being at least equal to the cross-section of the guides.

8. An introduction device according to any one of claims 2 to 6, wherein the transfer sleeve is provided with a concentrXc cylindrical channel having a diameter slightly exceeding the diameter of a sample holder so as to enable the central rod to introduce and maintain in place the sample holder, whose sample is intended for desorption in said concentric cylindrical channel, the latter having at its free end a cross-section slightly smaller than that of the sample holder, so as to form a stop for the sample holder in a desorption zone located within the ionization chamber or ion source, and, at its other end, a transverse groove extending in front of the introduction guide and the central guide, being open on the side of the introduction guide and having an end forming a stop in an extension of the concentric channel, as well as a first rectilinear side face facing the introduction guide and a second rectilinear side face, which is displaced with respect to the first side face, facing the discharge guide, said discharge guide being provided at its outlet with an inclined side face, the displacement between the inclined side face and the second rectilinear side face as well as the width of the transverse groove being at least equal to the cross-section of the guides.

9. An introduction device according to claim 7 or claim 8, wherein the transfer sleeve is provided on its rectilinear side face facing the introduction guide with a retractable stop in the form of a pushbutton or a deformable metal blade.

10. An introduction device according to claim 9, wherein th dir ct introduction rod has several introduction guides f r the sample holders In each of which is slidably mounted a supply rod, ach supply rod, as well as th discharg rod and the c ntral rod, being con-

trolled independently by a stepping m tor.

11. An introduction device according to any preceding claim, wherein it is inclined with respect to the horizontal plane and forms with the latter an angle (β) of at least 10°, the control mechanism being in a lower position with respect to the end of the direct introduction rod remote from said control mechanism and the introduction guide being located in the upper part of the rod above the central guide which is itself located above the discharge guide.

12. A process for the direct introduction of samples into a mass spectrometer performed by the device claimed in any one of the preceding claims, said process comprising the steps of successively introducing in a continuous or intermittent manner for a time interval which is pre-set or settable as a function of the pressure measured in the ion source by a programmable automaton or a microcomputer, several identical or different samples into the vicinity of the ionizing beam of the ion source of the mass spectrometer, whilst optionally maintaining the partial pressure of the product constant in the ion source by successive displacement of the sample holder with respect to the ion source and whilst optionally varying the desorption time and desorption temperature between individual samples.

13. A direct introduction process according to claim 12, wherein the process consists of performing the following operations under the control of the programmable automaton or microcomputer:

manual deposition of a sample, which is solid or in solution and is thermally stable or unstable, in an opening provided in each sample holder;

manual elimination of the solvent in the case of samples in solution;

manual introduction of the sample holders into an introduction guide of the introduction rod with the aid of a carrying gripper, the opening of each sample holder being oriented towards the transfer sleeve;

manual introduction of the free end of the rod into the ionization chamber through a lock;

heating of the desorption zone within the ionization chamber;

controlling the temperature;

heating or cooling the desorption zone until the desired temperature t_1 is obtained;

placing a first sample holder in the desorption zone; desorption in the vicinity of the ionizing beam of the ion source of the mass spectrometer for a time interval (dt);

optionally, checking the pressure in the ion source, the sample holder remaining in the desorption zone until the pressure t nds to d - crease;

removing th first sample holder;

checking the t mperatur ;

heating or cooling the desorption zone until either temp rature (t_1), or t mperature (t_2), which exceeds r is lower than (t_1), is obtained;

placing a second sample holder in the desorption zone;

continuing the operations until the final sample holder is placed in the desorption zone;

desorption of the final sample holder in the vicinity of the ionizing beam of the ion source of the mass spectrometer for a time interval (dt); and

removal of the final sample holder.

14. A direct introduction process according to claim 13, wherein in order to simultaneously place a sample holder and remove the preceding sample holder with respect to the desorption zone and in the case where an empty sample holder has previously been introduced into a discharge guide, the following stages are performed under the control of the programmable automaton or microcomputer:

starting a first stepping motor when the desorption of the sample in said preceding sample holder has taken place, said motor controlling a central rod which drops so that the sample holder is transferred in a transverse groove against the empty sample holder;

stopping the first motor;

starting up a second stepping motor controlling the translation of a supply rod which displaces the said sample holder, the latter also displacing the said preceding sample holder and taking up position facing an inclined end of the central rod, due to the simultaneous translation of a discharge rod, which enables the said preceding sample holder to enter the discharge guide;

stopping the second motor;

starting up the first motor controlling the central rod, which introduces the said sample holder into the channel of the transfer sleeve until coming into contact with a stop; and

stopping the first motor.

15. A direct introduction process according to claim 13, wherein in order to simultaneously place a sample holder and discharge a preceding sample holder with respect to the desorption zone, in the case when no empty sample holder has previously been introduced into a discharge guide, the following stages are performed under the control of the programmable automaton or microcomputer:

starting up a first stepping motor when the desorption of the sample in said preceding sample holder has taken place, said motor controlling a central rod which drops so that the sample holder drops by gravity in the discharge guide against the end of a discharge rod;

stopping the first motor when part of th end of the central rod is in a transvers groove;

starting up a s cond stepping motor controlling translation of a supply rod, which moves the sample holder to cause it to drop by gravity into the transv rse groove against

said end of the central rod, and translation of the discharge rod so as to form a space in said discharge guide making it possible to receive the sample holder after desorption;

stopping the second motor;

starting up the first motor controlling the central rod, which introduces the sample holder into the channel of the transfer sleeve until meeting a stop; and

stopping the first motor.

16. A direct introduction process according to claim 13, wherein for simultaneously putting into place a sample holder and discharging a preceding sample holder with respect to the desorption zone, in the case where no empty sample holder has previously been introduced into a discharge guide, the following stages are performed under the control of the programmable automaton or microcomputer:

starting up a first stepping motor when the desorption of the sample in the said preceding sample holder has taken place, said motor controlling a central rod which drops, so that the sample holder drops by gravity in the discharge guide against the end of a discharge rod, which then rises again until aligned with a rectilinear side face on the transfer sleeve;

stopping the first motor;

starting up a second stepping motor controlling translation of a supply rod, which moves the sample holder to cause it to drop by gravity into the transverse groove against the end of said groove, and translation of the discharge rod so as to produce a space in said discharge guide making it possible to receive the sample holder after desorption;

stopping the second motor;

starting up the first motor controlling the central rod, which introduces the sample holder into the channel of the transfer sleeve until meeting a stop; and

stopping the first motor.

17. A device for the direct introduction of samples into a mass spectrometer, the device being substantially as described herein with reference to the drawings.

18. A process for the direct introduction of samples into a mass spectrometer substantially as described herein with reference to the drawings.

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